# TECHNICAL STANDARD OPERATING PROCEDURE

Date: August 4, 1	<u> 1999</u>	SOP No	o. <u>MK-VBI70-05</u>					
Title: Sample Preparation								
APPROVALS:								
Author:	Morrison Knud	sen Corporation Date	e: August 4, 1999					
	vides procedures a laboratory analysis	and instructions for the preparation of soil sa	amples for on-site					
Received by QA I	<u>Unit</u>							
TEAM MEMBE	R	SIGNATURE/TITLE	<b>DATE</b>					
EPA Region 8	1	e Meht 10A Coordinator	8/5/99					
Morrison Knudsen Corp. Elle West 10A Coordinator 8/4/99								
REV.	DATE	REVISION DESCRIPTION	N					
1	8/4/99	Grinding of bulk soil prior to XRF analys	sis added					

#### 1.0 PURPOSE

The purpose of this procedure is to provide instructions to Morrison Knudsen personnel assigned to the VB/I-70 Project and their subcontractors for the preparation of soil samples.

#### 2.0 SCOPE

This procedure covers activities associated with preparation of soil samples for subsequent analysis by X-ray fluorescence spectrometry, inductively coupled plasma spectroscopy, and/or bioavailability tests.

#### 3.0 REFERENCES

Method 6200 Field Portable X-Ray Fluorescence Spectrometry For The Determination Of Elemental Concentrations In Soil And Sediment

Spectrace QuanX Laboratory X-Ray Fluorescence Analyzer Standard Operating Procedure

Standard Operating Procedure for Equipment Decontamination

Standard Operating Procedure for Waste Disposal

#### 4.0 **DEFINITIONS**

None

#### 5.0 RESPONSIBILITIES

The Sample Preparation Technician will be responsible for overseeing sample receipt and chain of custody both before and after the preparation process, and implementation of the sample preparation process.

The **Field Supervisor** will be responsible for quality and production of field laboratory operations.

The Site Health and Safety Officer will be responsible for verifying implementation of this procedure using safe laboratory practices.

The **Field Quality Assurance Coordinator** will be responsible for overseeing proper implementation of the quality control procedures, including tracking of blind standard performance evaluation samples, tracking of confirmation samples for off-site laboratory analysis, and specification of sample labels to be used for blind split samples.

The **Site Manager** will be responsible for ensuring that personnel are properly trained to this procedure.

### 6.0 REQUIREMENTS

#### 6.1 General

- 6.1.1 Sample preparation activities shall be performed only in areas designated for each activity.
- 6.1.2 Eating and smoking are prohibited in all areas of the sample preparation area.
- 6.1.3 Samples generally will be prepared in batches consisting of twenty field samples.

6.1.4 All non-dedicated equipment used during sample preparation must be decontaminated prior to use as described in the Decontamination SOP (MK-VBI70-07).

#### 6.2 Equipment

Sample drying trays

Permanent Marking pen

General purpose laboratory oven

#10 mesh stainless steel sieve

#60 mesh stainless steel sieve

Sample bags

XRF cups

Mylar

**Spatulas** 

Analytical balance accurate to 0.1 g, range of 0.1 g to 1000 g

Mortar and pestle, 140 mL or greater (or mill equipped with Burundum cylinders)

### 6.3 Soil Bulk Sieving

- 6.3.1 Select samples to be prepared. Prior to opening the sample bag, knead the contents to break up soil clumps and mix approximately two minutes or until the soil appears to be well homogenized. If the kneading process produces cohesive clumps, that observation will be noted in the Preparation Log.
- 6.3.2 Pour the sample from the bag onto a #10 sieve attached to a catch pan. Shake the sieve to pass the sample through the sieve into the catch pan. Using a gloved hand, break up other clumps that can be easily broken by hand and move soil to aid sieving. Dispose of any sample that did not pass through the sieve into the waste soil receptacle.
- 6.3.3 Pour approximately 6 ounces of the sieved sample into a pre-labeled (3-XXXXX-B) drying pan and pour the remainder of the sieved soil into a pre-labeled bag for archiving (3-XXXXX-A) under chain of custody documentation.

6.3.4 Document the date sieving was performed for each sample in the Sample Preparation Log Sheet.

## 6.4 Bulk Soil Drying

- 6.4.1 Set the oven temperature to 103-105 C (not to exceed 115 C).
- 6.4.2 Spread the sample on the drying tray in an even layer to promote even drying.
- 6.4.3 Check the oven temperature to verify proper temperature has been reached. Place the drying trays containing the samples into the oven(s). Leave the samples in the oven until completely dry as defined by a stable sample weight. Establish the drying time initially by recording weights for samples with varying soil moisture: 1) before drying, 2) at estimated completion, and 3) following an additional 15 minute drying time to confirm stable weight. Verify sample dryness for all samples by squeezing a portion of the sample between a gloved thumb and forefinger. Sample dryness is indicated by a lack of cohesiveness in the soil. Document the sample drying time for each sample on the Sample Preparation Log.
- 6.4.4 When samples are dry, remove from the oven and place in the ventilation area. **Before** placing samples at the ventilation area, verify that the blower is turned on.
- 6.4.5 Place the sample from the drying pan into a pre-labeled (3-XXXXX-B) sample bag. Completely seal the bag, then mix by turning the bag end over end slowly a minimum of ten times.

# 6.5 Bulk Soil Grinding and Cupping

- 6.5.1 Using a spatula, stir the contents of the sample bag thoroughly, then transfer 7 10 grams to the mortar.
- 6.5.2 Grind the soil using the pestle for ten minutes or until all material is evenly ground to a powder. If larger grained vegetation or soil materials remain, sieve the sample through a #60 sieve.
- 6.5.3 Using a spatula, fill the pre-labeled (3-XXXXX-B) XRF cup with soil from the mortar, filling cup ½ to ¾ full. Secure a piece of Mylar film over the top of the cup to seal.
- 6.5.4 Prepare XRF quality control samples as described in Section 6.7.2. Dispose of any unused ground soil into the waste soil receptacle.

## 6.6 Drying and Sieving Fine Fraction Soil

- 6.6.1 Selected archived bulk soil samples will be dried at a low temperature and sieved to isolate the naturally occurring fine fraction using a #60 mesh sieve.
- 6.6.2 Set the oven temperature to 45-48 C (not to exceed 50 C).
- 6.6.3 Pour approximately 8 ounces of soil onto a pre-labeled (3-XXXXX-A) drying tray and spread in an even layer to promote even drying. Return the remaining soil to the archive.
- 6.6.4 Check the oven temperature to verify proper temperature has been reached. Place the drying trays containing the samples into the oven(s). Leave the samples in the oven until completely dry as defined by a stable sample weight. Establish the drying time initially by recording weights for samples with varying soil moisture: 1) before drying, 2) at estimated completion, and 3) following an additional 15 minute drying time to confirm stable weight. Confirm sample dryness for all samples by squeezing a portion of the

sample between a gloved thumb and forefinger. Sample dryness is indicated by a lack of cohesiveness in the soil. Document the sample drying time for each sample on the Sample Preparation Log.

- 6.6.5 When samples are dry, remove from the oven and place in the ventilation area. **Before** placing samples at the ventilation area, verify that the blower is turned on.
- 6.6.6 Pour the dried sample onto a #60 sieve attached to a catch pan. Shake the sieve to pass the sample through the sieve into the catch pan. Dispose of any sample that did not pass through the sieve into the waste soil receptacle. Place the sample in the catch pan into a pre-labeled (3-XXXXX-F) sample bag. Completely seal the bag then mix by turning the bag end over end slowly a minimum of ten times. Using a spatula, stir the soil thoroughly and then fill the pre-labeled (3-XXXXX-F) XRF cup with soil from the sample bag, filling cup ½ to ¾ full. Secure a piece of Mylar film over the top of the cup to seal.

# **6.7 Quality Control Sample Preparation Procedure**

- 6.7.1 Sample preparation will be performed in an area separate from the XRF operations. The sample preparation technician who prepares sample batches containing blind quality control samples may not perform analysis on those samples. The XRF analyst will not observe the sample preparation and will not view the preparation logs in order to maintain sample anonymity to the analyst.
- 6.7.2 Prepare each of following quality control samples at rate of one per twenty field samples by filling two XRF cups with soil (following sample drying, sieving, mixing and grinding procedures):
  - One laboratory duplicate, labeled 3-XXXXX-B(or -F)D
  - One blind field split, labeled with a unique sample ID from the list of sample labels pre-assigned as blind split samples.

- 6.7.3 Prepare confirmation samples at a rate specified by the EPA Remedial Project Manager (initially one per three field samples) by transferring approximately 4 ounces from the prepared field sample bag (following bulk sieving, drying, fine sieving where applicable, and mixing; grinding of confirmation samples is not necessary) into a second bag and labeling the confirmation sample with the identical sample identification (i.e., 3-XXXXX-B or 3-XXXXXX-F). The confirmation sample will be submitted under chain of custody to an off-site laboratory for analysis by Method 6010B (ICP) as described in the Chain of Custody and Sample Handling SOP (MK-VBI70-02).
- 6.7.4 Document the laboratory duplicate in the "Notes" column of the Field Sample
  Preparation Log. Document the blind field split Sample ID and original Sample ID on the
  QC Data Sheet for Blind Soil Field Splits.
- 6.7.5 Prepare blind standards as directed by the Field Quality Assurance Coordinator by filling a pre-labeled XRF cup with soil from the blind standard sample provided. If the standard is not received pre-dried, sieved and ground, the sample will be prepared in accordance with sieving, drying, and grinding procedures detailed above in Section 6.3, 6.4 and 6.5. Label the cup with a unique sample ID from the list of sample labels pre-assigned as blind standards. Document the blind standard when prepared on the Performance Evaluation Standard (Blind Standard) QC Data Sheet.
- 6.7.6 Place 18 XRF cups for a single sample run into a staging container for transfer to the XRF Analyst (the analyst will complete the run with addition of a standard reference material and instrument blank).
- 6.8 Investigation Derived Waste Management
- 6.8.1 Remove the sample receptacle from under the ventilation hood and dispose of its contents into the waste soil drum when full and at the end of each day.

6.8.2 Place all non-dedicated sample drying trays, sieves, catch pans, and spatulas used during sample preparation in the receptacles for equipment decontamination.

### 7.0 ATTACHMENTS

Field Sample Preparation Logbook Sheet

QC Data Sheet, Blind Soil Field Splits

QC Data Sheet, Blind Performance Evaluation Samples

#### **ATTACHMENTS**

FIELD SAMPLE PREPARATION LOGBOOK SHEET

QC DATA SHEET, BLIND SOIL FIELD SPLITS

QC DATA SHEET, BLIND PERFORMANCE EVALUATION SAMPLES

Logbook DCN
-------------

# VBI70 Field Sample Preparation Logbook Sheet

			Sample Drying							Sieving				
Sample ID Prep Batch	Prep Batch	Confirmation	Date/Time Date/Time Drying Drying Begun <sup>b</sup> Completed <sup>b</sup>	Date/Time		Sample Mass <sup>c</sup> (grams)				Part	Particle Size Fraction <sup>d</sup>		Notes	
	Number Sample*	Sample*		Oven Temp (°C)	Before	After 1	After 2	After 3	Date Sieved	Raw Soil	Bulk (<2 mm)	Fine (<250 µm)		
													·	
								· ·						
						1								
			!	-										
			!									<del>-</del>		

a: Mark an "X" if a confirmation sample is prepared.

b' Enter Date in the following format: mm/dd/yy; Enter Time as 24-hour time (eg. 1340).

c: At least 2 measurements will be recorded. The sample is "completely dry" if the mass measurement is stable. A stability study will be performed as outlined in the Phase III Project Plan.

d :Mark an "X" for each sieve fraction collected

# VBI70 QC Data Sheet Blind Field Split Samples



Date	Sample ID	Sample Class	Original Sample	Prepared By	Notes
		BD			

# VBI70 QC Data Sheet Soil Performance Evaluation Standards

Date	Sample #	Sample	Lot No.	Certified Co	Prepared By	
- Z Odinpie #		Class	30.110.	Arsenic (ppm)	Lead (ppm)	
		PE				
		PE				
		PE				
		PE				
	·	PE				
		PE				
		PE				
······		PE				
	<u> </u>	PE				] 
		PE				ļ
	<u> </u>	PE				
<del></del>	<u> </u>	PE				
· · · · · · · · · · · · · · · · · · ·		PE				
	ļ	PE				
		PE				
		PE				
		PE			<del></del>	
· · · · · · · · · · · · · · · · · · ·		PE			<del></del>	
<del></del>		PE				
<del></del>		PE				
<u>.</u>		PE				,
<u></u> .		PE				
		PE				
		PE				
		PE		·		
		PE				
		PE				

Soil QC Sample Form: PE Samples

Page	